



February 6, 2017

To: Keith Nagel, Tom Barnett

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Subject: Interpretation of Sample Analysis from Initial Sheen investigation at AM Indiana Harbor West

Sample Descriptions:

Beginning on 1/10/2017 a sheen event was observed in the Indiana Harbor Ship Canal. High winds from the south and rain were the weather conditions. Soft and hard Booms present around IHW 009/010 as a standard spill control measure contained oil staining and it was observed that significant oil sheen was coming from upstream of ArcelorMittal in the Ship Canal. A sample of the oil buildup behind the boom was collected on 1/10/2017. This was assigned lab number 17A0422-1c by Microbac- CGL, which is ArcelorMittal IHW's contract Environmental Laboratory which provides analysis in support of the NPDES permit compliance.

The spill response contractor maintained the booms daily and the staining was variable but no sheen was observed coming out of 009/010. A Second sample of oil residue at the booms was pulled on 1/20/2017. This was assigned lab number 17A0422-3 by Microbac- CGL.

On 1/13/2017 USEPA came onsite with a Ponar dredge to attempt to collect sediment samples. This device has a spring mounted clamshell which can be triggered from the surface to collect sediment samples at depth. Three locations were attempted from the dock face. The first was downstream of the 009/010 discharge, this sample was silty but had no petroleum odor or oily appearance. The second was at the 009/010 outfalls and was gravel with no petroleum odor and no oil present. The third sample was upstream of 009/010 and was silty with a strong petroleum odor and an oil appearance. ArcelorMittal requested and received a split sample from this location. Microbac-CGL was directed by ArcelorMittal to perform a hexane extraction of the sediment and evaporate the hexane, leaving the oil component of the sediment. This was assigned lab number 17A0422-4 by Microbac- CGL.

Field observations:

Environmental Affairs while at the facility on 1/20/2017 and observed the various outfalls including the several upstream locations. Here are several important observations:

- At 009/010 diverter plates hangs down vertically several feet out from the dock face discharges. This was installed during the construction of the AM IHE to/IHW railroad lift bridge several years ago. With flow rates of 30 to 50 MGD each this cause's tremendous turbulence behind the diverter plate making sheen observations impossible at end of pipe. It also leads to scouring of the canal bottom- note the lack of silt from the Ponar dredge sample at the outfalls. One ongoing source of potential oil sheen may be when oily silt from the canal upstream moves into the scouring affect caused by the diverter plate.
- Upstream of the 009/010 outfalls, south of the railroad lift bridge there was significant oil sheen.

- At 001, while there was sheen present behind the weir plate it was not leaving our property and was not forming an oily buildup like the material at the 009/010 booms.
- Upstream of 001 on the other side of the railroad bridge south of 001 looking towards the south sheen was visible along the canal banks.
- At the Columbus Avenue bridge sheen, saturated soft booms and oil covered hard booms were visible along both banks and in both directions. While we were standing on the bridge, oil floated to the surface in the middle of the canal and became a large sheen circle within a couple of minutes.
- At the Indianapolis Avenue bridge saturated soft booms and oil covered hard booms were visible along both banks and in both directions

Analytical Methodology

The ability to identify the source of oil material in environmental media requires special consideration. Experience at an independent environmental laboratory for developing protocols that was very effective at identifying sources of oil in the environment and provided that capability to government and industrial clients were applied as described below.

Standard EPA methods utilized for NPDES compliance and Solid Waste analysis are of limited utility in this type of investigation because they are focused on specific compounds and quantifying them in the matrix. Oils are complex mixtures of base oils, which have many individual compounds, and additives that can also contain a variety of components. Also quantification is not the driver of a source investigation, it is the ability to obtain sufficient oil material from the sample location to allow for comparative analysis. . The petroleum industry has developed standard methods for oil fingerprinting that provide more useful comparative analysis. The most important of these is the FTIR scan. This provides an infrared spectrum "fingerprint" of the oil. Different types of oil have different fingerprints due to the base oils and additive packages used. The first phase of the investigation is to extract the oil fraction from the environmental matrix and generate the FTIR fingerprint. A general interpretation of the nature of the oils can be made. The next step is to overlay potential source FTIR fingerprints to narrow the field of possible sources based on similarity of fingerprint. More specific analysis targeting additives or other marker compounds can be selected to refine the dataset. The attached Analytical report # 17A0572 by Microbac is the initial FTIR fingerprints of the three samples described at the beginning of this Memo. Microbac Merrillville prepared the samples and the Micobac Forensic lab in Boulder CO performed the FTIR analysis.

The following is a discussion of the scans presented in the report.

Figure 1 on the top of page 3 of the report shows the oil fraction of the sediment from upstream of 009/010 compared with a laboratory reference IR spectrum of paraffinic base oil (no additives). The absorbance peaks at 3000 cm^{-1} and $1500\text{ to }1400\text{ cm}^{-1}$ are the basic petroleum oil fingerprint. It is important to note that there are a significant number of additional peaks beginning at 1800 cm^{-1} and extending to 700 cm^{-1} . These are potential additives or contaminants in the extracted sample.

Figure 2 on the bottom of page 3 of the report shows the oil collected from the boom at 009/010 on 1/20/2017 compared with a laboratory reference IR spectrum of paraffinic base oil (no additives). The absorbance peaks at 3000 cm^{-1} and 1500 to 1400 cm^{-1} are the basic petroleum oil fingerprint. It is important to note that there are a significant number of additional peaks beginning at 1800 cm^{-1} and extending to 700 cm^{-1} . These are potential additives or contaminants in the extracted sample.

Figure 3 on the top of page 4 of the report shows the oil collected from the boom at 009/010 on 1/10/2017 and 1/20/2017 compared with oil fraction of the sediment from upstream of 009/010. A critical point to note that the peak patterns from 1800 cm^{-1} and extending to 700 cm^{-1} show a high degree of match. Peak heights can vary due to amount of oil available to scan, however the wavelength locations of the peak pattern are consistent among all three.

The laboratory also notes at the bottom of figure 3 that the sharp peak at 700 cm^{-1} may indicate the presence of chlorinated hydrocarbons. In a mineral oil that is typically a chlorinated paraffin. ArcelorMittal uses no lubricants containing chlorinate paraffin. Given that the location of the sediment sample that generated the oil fraction lab # 17A0422-03 was upstream of 009 and 010 and the fact that ArcelorMittal uses no lubricants with chlorinated paraffin the source of oil sampled in the booms on 1/10/2017 and 1/20/2017 is most likely historical oily residue in the canal being stirred up by weather.

Next Phase of Analysis:

An intermittent sheen event lasting about 10 minutes was observed at 009/010 on 1/25/2017. There have been more samples collected both within ArcelorMittal and upstream of ArcelorMittal in the canal and those samples are being processed. ArcelorMittal is also working with our Lubricant supplier Shell Oil to provide technical assistance.